DETERMINATION OF THE SPECIFIC SURFACE OF ACTIVATED CHARCOAL BY ADSORPTION FROM SOLUTION

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The wide use of activated charcoal in many branches of industry (rubber, varnish coloring, polygraphy) has made it desirable to investigate its adsorptive properties and to find a rapid and dependable method of determining its specific surface.

In [1] and [2] the possibility was pointed out of using adsorption from solution as a rapid method of estimating the specific surface S on the basis of a known absolute* adsorption isotherm. When the amount x adsorbed by one gram of the adsorbent in some equilibrium concentration C has been determined experimentally, and the absolute magnitude for adsorption Γ has been obtained from the absolute isotherm for the same concentration C then

$$\Gamma = \frac{x}{S} . \qquad (1)$$

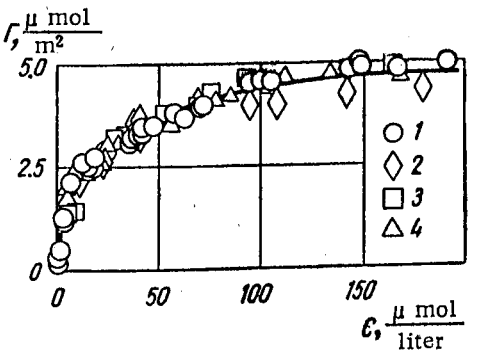


Fig. 1. Absolute values of the adsorption of phenol from a solution in water by charcoal; 1) Ukhta channel black; 2) graphitized Ukhta channel black; 3) Ukhta thermal; 4) Sferon-6.

It is usual to express x in mmol/g and Γ in μmol/m^2 in which case

$$S = \frac{1000\, x}{\Gamma} \text{ m}^2/\text{g} \qquad (2)$$

Here x and Γ representing the amounts of the adsorbable substance in the surface layer of the adsorbent are both taken for the same equilibrium solution. If the absolute adsorption isotherm for the system phenol-water-activated charcoal is available, the same method of measurement used earlier by Kaunowski and Hofmann [4] can be applied to give a very simple, rapid, and reliable technique for determining the specific surface using one or two values of the adsorption. We determined absolute isotherms of adsorption of phenol from a water solution for three different types of activated charcoal: Ukhta thermal, Ukhta channel black, and Ukhta channel black subjected to graphitization at 1700°. It is evident from Fig. 1 that the absolute adsorption isotherms of phenol in a water solution for the different types of activated charcoal can be given by one curve. This makes it possible to use formula (2) to determine the specific surface of activated charcoal from various sources. For this determination one or two weighed samples of 1.0-1.5 g of charcoal are put in ampoules and, using a calibrated pipette, 7-10 cc is added of a solution of phenol in water of known concentration (about 200 mmol/liter) which has been prepared earlier in a measuring flask. The ampoules are then sealed or corked and either placed in a thermostat with a device for shaking the sample at 20°, or simply in a shaker (if the room temperature does not vary more than a few degrees). After the solution has been shaken for about 15 min, it is drawn off by a pipette and its equilibrium concentration C is determined.

If an ITR-2 interferometer is used for measuring the concentration, a calibration curve must first be drawn. First of all the zero reading of the instrument is found. In order to do this both sections of the cell of the interferometer are filled with water, the cell is placed in the water thermostat of the apparatus, and after the

*For plotting the absolute adsorption isotherm the measured adsorption values are calculated per square meter.

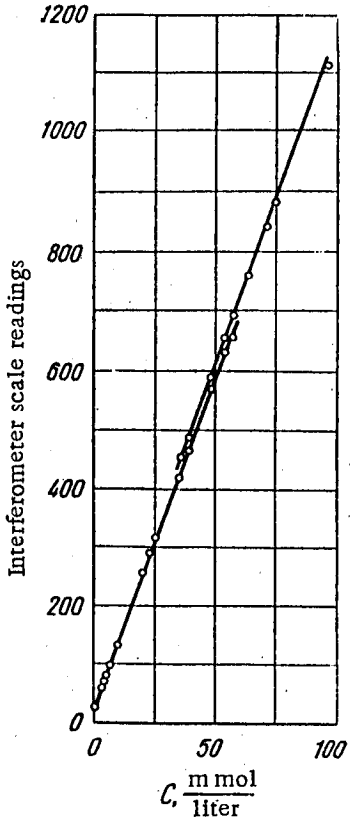


Fig. 2. Interferometer calibration curve for solutions of phenol in water at 20°.

TABLE

Interpolated Data for Drawing the Absolute Isotherm for Adsorption on Activated Charcoal of Phenol from a Solution in Water

Equilib. conc. C, mmol/liter	Abs. magn. of adsorption Γ, μmol/m²	Equil. conc. C, mmol/liter	Abs. magn. of adsorption Γ, μmol/m²
5	1.50	100	4.40
10	2.10	110	4.40
20	2.75	120	4.50
30	3.15	130	4.50
40	3.45	140	4.55
50	3.70	150	4.60
60	3.85	160	4.60
70	4.00	170	4.65
80	4.25	180	4.70
90	4.35	190	4.70

illuminator has been switched on the spectrum is observed in the eye-piece. Two spectra are visible in the interferometer; the movable one formed from light that has passed through both sections of cell and the fixed one formed from the ray that has traversed the lower section only (the comparison spectrum). The drum of the compensator is turned until the two spectra coincide. The zero reading obtained on the scale is written down. After this the water in one of the sections is replaced by a series of several previously prepared solutions of known concentration up to 200 mmol/liter, and the readings corresponding to each solution are recorded. The readings obtained are plotted on a graph as in Fig. 2. In order to use the calibration curve, it is necessary to note the cell number and to stipulate that one of the sections of the cell, e.g., the right, be always used for the solvent, and the other, the left, be always used for the solution. All readings must be taken for the same position of the cell. For the analysis of the equilibrium solution the suspended charcoal in the specimen in the ampoule is allowed to settle, and the solution is then placed in the right section of the interferometer cell. A reading is taken on the scale and by referring to the calibration curve the concentration corresponding to this reading is obtained. As can be seen in Fig. 2 there exists a range of concentration where two scale readings correspond to one and the same concentration. This must be taken into account in drawing the calibration curve and in its use [5].

In the absence of an interferometer, the following method of analysis of the equilibrium solution is recommended [6]. To 5 ml of the solution of phenol in water is added 20 ml of bromine water and the mixture is thoroughly shaken for 5-10 min. The excess bromine water is neutralized by 2-3 g of calomel according to the reaction

$$C_6H_5OH + 4Br_2 \rightarrow C_6H_2Br_3OBr + 4HBr$$
$$Br_2 + Hg_2Cl_2 \rightarrow HgBr_2 + HgCl_2.$$

The excess calomel is filtered out and the precipitate is washed with distilled water. Two or three drops of methyl red are added to the filtrate which is then titrated against a 0.15 N solution of alkali. Good results can be obtained with concentrations up to 120 mmol/liter. The original concentration C_0 of the solution added to the charcoal in the ampoule is easily calculated from the weight of the phenol used, which is chosen so that the concentration of the solution after adsorption does not exceed 120 mmol/liter. For charcoal with specific surface

less than 50 m^2/g (thermal, for example) C_0 should be between 130 and 150 mmol/liter and for charcoal with a specific surface more than 50 m^2/g, C_0 should be between 150 and 200 mmol/liter.

When the concentration of the equilibrium solution has been determined, the adsorption x is obtained by using

$$x = \frac{(C_0 - C)V}{1000\ m}, \tag{3}$$

where C_0 and C are the concentrations of the original and the equilibrium solutions, m is the weight of the adsorbent in grams, and V is the volume of solution in the ampoule in ml. Reference to the absolute isotherm will give the absolute value of the adsorption Γ for the concentration of the equilibrium solution and the value of the specific surface S can then be obtained from formula (2). The accompanying table supplies the data necessary for drawing the absolute isotherm for adsorption on activated charcoal of phenol from a water solution.

The time necessary for the determination of specific surface by this method depends basically on the time taken to reach adsorption equilibrium. Investigations into the kinetics of the adsorption of phenol on Ukhta channel black charcoal from a solution in water showed that after 15 min of shaking, the deviation from equilibrium was about 10%, and at the same time the experimental error was about 2% for these concentrations. Stable equilibrium was attained only after 15 hr. The time of shaking can therefore be related to the accuracy of the determination of the surface. Even in the case where it is necessary to continue the experiment to the stage where a stable equilibrium is attained, no time should be wasted on any specimen, since with this method work can proceed simultaneously on a series of specimens and since one original solution may be prepared beforehand in large quantities for use with them all.

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PHASE ANALYSIS BY MEANS OF X-RAY DIFFRACTOMETERS

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The use of x-ray devices with registration of x-rays by ionization (diffractometers) considerably widenes the scope and possibilities in the application of x-ray quantitative and qualitative phase analysis [1]. The methods of quantitative and qualitative phase analysis by means of Soviet -manufactured URS-501 diffractometer are described in the present work [2].

Qualitative Phase Analysis

The qualitative phase analysis is based on an accurate determination of the location of diffraction maxima